Data Validation Report

TDD No: 09-04-01-0011
PAN: 001275.0440.01TA
Site: El Dorado Hills
Laboratory: Lab/Cor, Inc.
Reviewer: Denise A. Shepperd, Trillium, Inc.
Date: January 27, 2005

I. Case Summary

SAMPLE INFORMATION:

Asbestos Samples: CC6-H6-1CP-100704; CC6-H6-2CP-100704; CC6-L6-1CA-100704; CC6-L6-1CB-100704; CC6-L6-2CB-100704; CC6-L6-3CB-100704; CPS-H2-1FD-100704; CPS-H2-1ZB-100704; CPS-H2-2FD-100704; CPS-H2-4FD-100704; CPS-L2-15CH-100704; CPS-L2-1CH-100704; CPS-L2-1NA-100704; CPS-L2-2CH-100704; CPS-L2-3CH-100704; CPS-L2-4CH-100704; CPS-L2-5CH-100704; JOGB-L2-12AD-100704; JOGB-L2-1AD-100704; JOGB-L2-2AD-100704; JOGB-L2-3AD-100704; JOGB-L2-4AD-100704; JOGB-L2-5AD-100704; NYB-H2-1FD-100704; NYB-H2-1ZP-100704; NYB-H2-2FD-100704; NYB-H2-4FD-100704; NYB-L2-11CH-100704; NYB-L2-1CH-100704; NYB-L2-1NA-100704; NYB-L2-1ZB-100704; NYB-L2-2CH-100704; NYB-L2-3CH-100704; NYB-L2-4CH-100704; NYB-L2-5CH-100704; CC9-L6-11CA-101004; CC9-L6-1CA-101004; CC9-L6-1CC-101004; CC9-L6-2CC-101004; CC9-L6-3CC-101004; JEB-H1-6FD-101004; JEB-H1-7FD-101004; JEB-L2-1CH-101004; JEB-L2-1NA-101004; JEB-L2-2CH-101004; JEB-L2-3CH-101004; JEB-L2-4CH-101004; JEB-L2-5CH-101004; JEG-L2-15CH-101004; JEG-L2-1AD-101004; JEG-L2-1CH-101004; JEG-L2-1ZB-101004; JEG-L2-2CH-101004; JEG-L2-3CH-101004; JEG-L2-4CH-101004; JEG-L2-5CH-101004; JEG-L2-FB-101004; JEP-L2-1CH-101004; JEP-L2-1NA-101004; JEP-L2-1ZB-101004; JEP-L2-2CH-101004; JEP-L2-3CH-101004; JEP-L2-4CH-101004; JEP-L2-5CH-101004; TRA-L2-1ZP-100904; CPS-H2-14FD-100704; CPS-H2-1PG-100704; CPS-H2-2PG-100704; CPS-H2-3FD-100704; CPS-H2-3PG-100704; CPS-H2-4PG-100704; CPS-H2-5FD-100704; CPS-H2-5PG-100704; CPS-L2-FB-100704; JEB-L2-1ZP-101004; JOGB-H2-12TR-100704; JOGB-H2-1TR-100704; JOGB-H2-2TR-100704; JOGB-H2-3TR-100704; JOGB-H2-4TR-100704; JOGB-H2-5TR-100704; NYB-H2-12FD-100704; NYB-H2-13PG-100704; NYB-H2-1PG-100704; NYB-H2-22FD-100704; NYB-H2-2PG-100704; NYB-H2-3FD-100704; NYB-H2-3PG-100704; NYB-H2-4PG-100704; NYB-H2-5FD-100704; NYB-H2-5PG-100704; NYB-L2-1ZP-100704; TRA-H2-1ZP-100904; TRA-H8-11TR-100904; TRA-H8-1TR-100904; TRA-H8-2TR-100904; TRA-H8-3TR-100904; TRA-H8-4TR-100904; TRA-H8-5TR-100904; TRA-L2-1ZB-100904; JEB-H1-12FD-101004; JEB-

H1-1FD-101004; JEB-H1-2FD-101004; JEB-H1-3FD-101004; JEB-H2-4FD-101004; JEB-H2-5EFD-101004; JEB-H2-5WFD-101004; JEG-H2-1FD-101004; JEG-H2-2FD-101004; JEG-H2-3FD-101004; JEB-H2-4FD-101004; JEG-H2-5FD-101004; JEP-H2-15FD-101004; JEP-H2-1FD-101004; JEP-H2-2FD-101004; JEP-H2-3FD-101004; JEP-H2-4FD-101004; JEP-H2-5FD-101004; JOGB-L2-1NA-100704

Matrix:	119 Air samples
Analysis:	Asbestos by Transmission Electron Microscopy
Collection Dates:	October 7 through 10, 2004
Sample Receipt Date:	October 12, 2004
Analysis Date:	October 12, 2004, through January 1, 2005
Analytical Method:	ISO Method 10312

FIELD QC:

Field Trip Blanks (TB):	CPS-H2-1ZB-100704; NYB-L2-1ZB-100704; JEG-L2-1ZB-101004; JEP-L2-1ZB-101004; TRA-L2-1ZB-100904
Filter Blanks (FB):	None
Equipment Blanks (EB):	None
Background Samples (BG):	None
Field Duplicates (D1):	Not Identified
Performance Evaluation Samples (PE):	NYB-H2-1ZP-100704; TRA-L2-1ZP-100904; JEB-L2-1ZP-101004; NYB-L2-1ZP-100704; TRA-H2-1ZP-100904

TABLES:

1A:	Analytical Results with Qualifications
1B:	Data Qualifier Definitions for Inorganic Data Review

SAMPLING ISSUES:

Six chain of custody (COC) documents were included in the data package and were properly completed except that only the first of these documents was signed as relinquished to FedEx for shipment. An airbill number was recorded on this page also. All six COC records were appropriately signed and dated on receipt at the laboratory. Each page of the COC record should be signed, dated, and initialed each and every time custody of the samples represented therein is transferred.

The COC records included all of the field samples in the data package, as well as many additional samples.

VALIDATION PARAMETERS AND COMMENTS:

I. Holding Times, Preservation and Sample Integrity

This parameter is evaluated to ensure that sample custody is documented from collection through analysis, samples are analyzed within the recommended holding time, and that no alteration in sample content has occurred during sample shipment, handling, and storage.

There is no established holding time or storage condition for asbestos samples.

II. Calibration

The analyses of materials of known content ensures that identification and quantitation of analytes will be accurate for all samples. Review of the documentation provided for appropriate calibration determines whether or not the analytical results reported by the laboratory are valid and supported by the data.

The data deliverables for this project were included in multiple data packages. The calibration documentation was provided in a single separate package associated with all of the site sample data packages.

A letter representing documentation of an NVLAP laboratory site assessment conducted on 11/7/03 was included in the calibration data package. The letter included (dated 5/10/04) indicated that the laboratory met the on-site assessment requirements.

Results and evaluator notes and tables were included for an NISTIR 5351 analysis of an inter-laboratory QC sample. The laboratory's raw data were compiled and assessed by Batta Labs. Analysts were identified by initials and included all of the initials documented with this sample set, except "TM" and "JH." According to the assessor's notes, the sample included chrysotile fibers and structures and the laboratory's results were within NVLAP and NISTIR 5351acceptance limits.

Results for a New York State Department of Health Environmental Laboratory Approval Program proficiency test, conducted between 9/7/04 and 11/9/04, were included. The proficiency samples included asbestos in air. The laboratory's results were satisfactory for all four of the air sample categories. Actinolite and amosite fiber types were identified and counts were acceptable according to the data sheet. No raw data were provided for this proficiency sample. Upon request, these data were provided by the laboratory on 1/26/05 and inserted into the QC data package.

Acceptable instrument calibration was documented in the data package, including screen and camera magnification, camera length and camera constant, spot size, k-factor, beam dose, EDS sensitivity and peak intensity. No documentation of grid opening size was provided. Documentation was provided in the separate proficiency and calibration data package for October through December, 2004, for both of the instruments used for analysis of samples included in this data package. Analyses of the samples in this data set were performed during this time period.

Based on the fact that the laboratory demonstrated proficiency in the performance evaluation (PE) analyses performed in the third quarter of 2004, and that these PE samples included the two predominant asbestos types detected in this field sample set, no action was taken by the validator. It is recommended however, that supporting data be expanded to include raw data supporting the identification of all asbestos types detected in PE samples and demonstration, wherever possible, of the correct identification (in known reference materials) of all fiber types detected in a field sample set.

III. Blanks

Sample matrices known to be devoid of the analytes of interest (method blanks) are prepared and analyzed with each analytical batch. Evaluation of this parameter ensures that contamination introduced during preparation and analyses is not attributed to the field samples.

Other blanks may be generated in the field or laboratory to ensure that no contamination is introduced during sampling and/or storage.

Blanks required for this project included Filter Blanks and Field Trip Blanks. Five field trip blanks (CPS-H2-1ZB-100704; NYB-L2-1ZB-100704; JEG-L2-1ZB-101004; JEP-L2-1ZB-101004; TRA-L2-1ZB-100904) were included with this sample set. Field trip blank TRA-L2-1ZB-100904 was rejected at the preparation step due to a hole in the filter. This blank was not reported in the data set. No asbestos structures were detected in any of the four remaining field trip blanks. No filter blanks were included with this site sample set.

IV. Spiked Samples

The analytes of interest are added in known concentrations to like-matrix blanks or authentic field samples before preparation. This parameter is evaluated in order to assess the laboratory's ability to preserve and recover the compounds of interest.

The analytical method does not require laboratory spiked sample analyses. It is recommended by the validator that some type of laboratory prepared or purchased spiked analyses be performed with each analytical sample batch.

The project requirements specified that results from the most recent inter-laboratory study would be acceptable as an LCS sample for these data. This requirement was met by the laboratory and reported results for the inter-laboratory study sample were acceptable for all air sample parameters (see Section I).

Five of the samples (NYB-H2-1ZP-100704; TRA-L2-1ZP-100904; JEB-L2-1ZP-101004; NYB-L2-1ZP-100704, and TRA-H2-1ZP-100904) analyzed with this set are performance evaluation (PE) samples submitted along with the field samples. Two of these samples (NYB-H2-1ZP-10074 and JEB-L2-1ZP-10104) were reported by the laboratory to contain actinolite, chrysotile, and tremolite fibers with total asbestos structure counts of 108 and 120 (equivalent to 0.184 and 1.33 structures per mm^2 in air, respectively).

The three remaining samples (TRA-L2-1ZP-100904; NYB-L2-1ZP-100704, and TRA-H2-1ZP-100904) were reported by the laboratory to contain chrysotile and tremolite with total asbestos structure counts of 101 to 105 (equivalent to 1.27, 1.13, and 0.332 structures per mm^2 in air, respectively).

True values were not supplied for these PE samples; therefore no assessment of laboratory performance could be made.

V. Duplicate/Replicate Samples

Results for duplicate/replicate samples are evaluated to assess the laboratory's precision for the analytes of interest in the applicable sample matrix. For asbestos analyses, duplicate and replicate measurements take the form of a combinations of variables which include the preparation of the grid, the choice of grid openings to be analyzed, and the analyst performing the counting and identification of structures.

The laboratory included all of the QC samples from all of the field sample sets in a separate data package under a separate report number (041176R1).

The two analysts, JH and TM, not represented in the PE sample analyses included with the data packages for this project (see Section I) did perform intra-laboratory replicate and duplicate analyses on associated field samples. Results for these QC analyses for both analysts were within the sample-specific acceptance limits.

The quality assurance project plan (QAPP) requires five types of laboratory duplicate/replicate analyses, each to be performed at a rate of 5% (one for every twenty) of the field samples. Based on the 90 field samples reported in this data package, five of each of these QC sample pairs were required. The laboratory compared the primary asbestos structure count for each of the QC samples prepared and analyzed. Results for all of the duplicate/replicate pair types were evaluated based on 95% confidence limits determined from the original sample count result. Results for all of the reported QC samples were within the laboratory's calculated limits. A summary of the laboratory QC samples included with this data set are as follows:

Replicate analyses:

- one sample, CPS-H2-4FD-100704, was analyzed as a replicate wherein the same analyst counted different grid openings;

- two samples, CPS-H2-4FD-100704 and JEB-H1-6FD-101004, were analyzed as replicates wherein a different preparation was analyzed by the same analyst;

Duplicate analyses:

- three samples, CPS-H2-4FD-100704, CPS-H2-14FD-100704, and JOGB-H2-5TR-100704, were analyzed as duplicates wherein the same grid openings were recounted by a different analyst;

- one sample, JOGB-H2-5TR-100704, was analyzed as a duplicate wherein different grid openings were counted by a different analyst; and

- four samples, CC6-L6-1CA-100704, CPS-H2-4FD-100704, JEB-H1-6FD-101004, and CPS-H2-1PG-100704 were analyzed as duplicates wherein a different analyst counted a new preparation.

Five or more QC samples should have been included for each of these five QC sample categories in order to satisfy the 5% requirements of the QAPP.

One additional type of QC sample, not identified by the QAPP, was included. Samples CC6-H6-2CP-100704, CC6-L6-1CA-100704, and CPS-H2-1PG-100704 were recounted by the same analyst, counting the same grids.

According to the QAPP provided with the data packages, field duplicates were required at a rate of 10% of field samples. Field duplicate pairs were not identified or evaluated as part of this validation effort.

VI. Identification

Identification of asbestos structures and fibers is dependent on sample preparation techniques, analyst training, instrument operation, and data interpretation. Comparison with results from known standards is used to evaluate the accuracy of the structure identification for field samples.

Chrysotile, actinolite, edenite, and tremolite were reported by the laboratory in the field samples. According to the report forms provided in the separate QC package, the laboratory correctly identified actinolite, chrysotile, and amosite in PE sample analyses performed in the third quarter of 2004. Comparison of identification between the various analysts, grid opening, and preparations combinations that make up the daily QC for these analyses were acceptable. Therefore; based on the documentation provided, fiber and structure identifications for chrysotile and

actinolite were determined to be valid as reported. It was assumed that the laboratory correctly identified the other amphibole structures that were reported in the field samples.

VII. Quantitation and Reported Detection Limits

Raw data documentation is reviewed to ensure that all reported results and detection limits are correctly calculated, accurately reported, and supported by the raw data.

With a few exceptions, results for asbestos categories, fiber density, and detection limits were correctly calculated and accurately reported by the laboratory and could be verified by the validator, using the information included on the reporting forms and the chain of custody records. The following exceptions were noted:

The report form for sample CPS-L2-1CH-100704 lists 90 grid openings counted. According to the raw data count sheet, 91 openings were counted. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

The dimensions and aspect ratio for a fiber listed on the raw data count sheet for sample CPS-L2-4CH-100704 were not recorded. Counts for individual categories associated with this fiber could not be verified but there is no effect on the total structure counts for this sample.

The count sheet for sample JOGB-L2-3AD-100704 described grid number B31 as "film doubled up over entire grid." As a result this grid is not acceptable for counting and should be deleted from the count. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

The count sheet for NYB-L2-2CH-100704 listed 90 grid openings counted whereas the report form lists 89. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

The count sheet for sample CPS-H2-1PG-100704 lists 1 grid as "cracked" and 1 grid as "torn." Neither of these grid locations should be included in the count, reducing the grids counted from 23 to 21. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

An actinolite bundle listed on the count sheet appears to have been overlooked from the count of total asbestos fibers and bundles in samples CPS-H2-4PG-100704 and CPS-H2-5FD-100704. On the count sheet for JEB-L2-1ZP-101004 two bundles, one tremolite and one chrysotile, appear to have been omitted from the asbestos fiber and bundle count. The validator applied a "J" qualifier to the results for the asbestos fiber and bundle category for these three samples. The total and primary asbestos structure counts are not affected.

Five field samples (NYB-L2-3CH-100704, NYB-H2-3FD-100704, JEG-H2-1FD-101004, JEG-H2-2FD-101004, and JEG-H2-5FD-101004) were rejected by the laboratory at analysis, due to high particulate loading of the filters, and these samples were not analyzed with this data set.

Twenty samples (CC6-L6-1CB-100704, CC6-L6-2CB-100704, CC6-L6-3CB-100704, NYB-H2-1FD-100704, NYB-H2-2FD-100704, NYB-L2-11CH-100704, NYB-L2-1CH-100704, NYB-L2-4CH-100704, NYB-L2-5CH-100704, CC9-L6-11CA-101004, CC9-L6-1CA-101004, CC9-L6-CC2-101004, CC9-L6-3CC-101004, JEG-L2-15CH-101004, JEG-L2-1AD-101004, JEG-L2-1CH-101004, JEGL2--2CH-101004, JEG-L2-3CH-101004, JEG-L2-4CH-101004, and JEG-L2-5CH-101004) were rejected at the sample preparation step due to loose particulate on the filter.

Samples TRA-L2-1ZB-100904 and JEB-H1-12FD-101004 were rejected at the preparation step because there were holes in the filters.

Sample JEB-H1-3FD-101004 was rejected at the preparation step due to filter defects that obstructed airflow.

Sample NYB-H2-12FD-100704 was rejected at preparation due to loose particulate and possible moisture on the filter.

VIII. System Performance

This parameter is evaluated to ensure that the laboratory analytical systems were functioning properly at the time of analyses and that methodology appropriate to the analyses were followed.

The analytical systems appear to have been working satisfactorily and to have been calibrated properly at the time of these analyses, based on the available documentation.

IX. Documentation

Data and documentation completeness is critical in providing support for the reported results. Problems encountered with the nature or quality of the data package documentation are addressed.

Tape had been applied to the count sheet for CC6-L6-1CA-100704 and written over, obscuring the information underneath and preventing verification of the counts for the affected fibers and/or structures.

The QC summary form listed 6 as the primary structure count result for the original sample analysis of JOGB-H2-5TR-100704. According to the sample raw data and report form the primary structure count result for this sample is 5 and the acceptance limits should be calculated from this value instead of 5. The validator made the appropriate corrections to the QC summary form in the data package.

The QC summary form lists the parent sample for analysis of QC sample 041176-29 as JEB-H1-6FD-10104 whereas the raw data for this QC sample indicate that the parent sample is CPS-H2-4FD-100704. At the data user's discretion, the laboratory should be contacted to request the true identification of this QC sample.

The TEM negative number 1051 was listed on the count sheet for an actinolite fiber counted at grid location B23 C13 in sample JOGB-H2-3TR-100704. The negative included in the data package is number 1052. The validator edited the count sheet to reflect the correct identification.

No raw data were provided in the data package for the proficiency samples analyzed in support of the laboratory's accreditation. Raw data to support the identification of actinolite and amosite were received upon request on 1/26/05.

Raw data for chrysotile fibers were not included in the data package for review. Raw data documenting fiber identification for the other asbestos types identified in the field samples were present in the data package. Upon request representative raw data for chrysotile from each sample set were supplied by the laboratory on 1/27/05.

The analysis date listed on the printouts for the EDD for some of the field samples is Jan 1, 1997.

TEM negative number 1079 and EDS number 794 were included in the data package. These raw data are associated with sample JEB-H1-3FD-101004 which was rejected due to filter defects. The validator removed these raw data from the data package.

Count sheets included in the data package are computer generated forms. No date of the actual count is presented on these forms. If there is a corresponding bench sheet from which these forms are prepared, these should be supplied as a part of the data package. Alternately, the count forms could be modified to include analyst initials and date of analysis.

The legend for the count sheets, which defines the codes used for the structure counts, lists PSCH as the code for protocol chrysotile structures. The code actually appearing on the count sheets for this category is PCAS.

Raw data are an integral part of a complete and defensible data package. Edits made on all data should be performed correctly. Proper editing requires drawing a single line through the incorrect information, adding the correct information, and initialing and dating the changes.

Asbestos structures identified in the field samples included actinolite, chrysotile, and tremolite. Examples of known materials included in the data package in support of the sample analyses included only actinolite, chrysotile, and amosite, identified in the proficiency sample analyses. Based on the data provided for validation, the identification of the other fiber types in a known standard was not documented.

COMMENTS:

A. Tape had been applied to the count sheet for CC6-L6-1CA-100704 and written over, obscuring the information underneath and preventing verification of the counts for the affected fibers and/or structures.

B. The report form for sample CPS-L2-1CH-100704 lists 90 grid openings counted. According to the raw data count sheet, 91 openings were counted. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final reports.

C. The dimensions and aspect ratio for a fiber listed on the raw data count sheet for sample CPS-L2-4CH-100704 were not recorded. Counts for individual categories associated with this fiber could not be verified but there is no effect on the total structure counts for this sample.

D. The raw count sheet for sample JOGB-L2-3AD-100704 described grid number B31 as “film double up over entire grid.” As a result this grid is not acceptable for counting and should be deleted from the count. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

E. The count sheet for sample CPS-H2-1PG-100704 lists 1 grid as “cracked” and 1 grid as “torn.” Neither of these grid locations should be included in the count, reducing the grids counted from 23 to 21. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

F. The count sheet for NYB-L2-2CH-100704 listed 90 grid openings counted whereas the report form lists 89. The validator recalculated the concentrations, analytical sensitivity, and detection limits for this sample and made the appropriate corrections to the final report.

G. An actinolite bundle listed on the count sheet appears to have been overlooked from the count of total asbestos fibers and bundles in samples CPS-H2-4PG-100704 and CPS-H2-5FD-100704. On the count sheet for JEB-L2-1ZP-101004 two bundles, one tremolite and one chrysotile, appear to have been omitted from the asbestos fiber and bundle count. The validator applied a “J” qualifier to the results for the asbestos fiber and bundle category for these three samples on the Analysis Detail reported in the data package and on the originally received electronic data deliverables (EDD) tables. The total and primary asbestos structure counts are not affected.

ADDITIONAL COMMENTS:

Results for samples in this data set were determined to be valid as reported by the laboratory with the exceptions noted above. No qualifiers were added to the primary and total asbestos structure results by the validator.

Results for asbestos structure >5 : m and asbestos fibers and bundles >5 : m were qualified as estimated (J). These qualifiers appear only on the Analysis Detail reports included in the data package and in the originally provided electronic data deliverables (EDD) tables.

Reported results, analytical sensitivity, and detection limits are considered to be accurate within the bounds of the 95% confidence limits determined for each sample.

The data results tables included as Table 1A include only the primary and total asbestos structure counts. Counts for individual categories required by the project Scope of Work are presented in the associated electronic data deliverables (EDD) tables.

This report was prepared according to the specifications of the analytical method, ISO Method 10312 " Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method," the document "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94, and Trillium, Inc.'s SOP No. 0497-06A, for Validation of Analytical Data: Inorganic Analytes.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared in accordance with the document, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94.

U The analyte was analyzed for, but was not detected above the level of the reported value. The reported value is either the sample quantitation limit or the sample detection limit.

L Indicates results which fall between the sample detection limit and the CRDL. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.

J The associated value is an estimated quantity. The analyte was analyzed for and was positively identified, but the reported numerical value may not be consistent with the amount actually present in the environmental sample.

R The data are unusable. The analyte was analyzed for, but the presence or absence of the analyte cannot be verified.

UJ A combination of the “U” and “J” qualifier. The analyte was analyzed for but was not detected. The reported value is an estimate and may be inaccurate or imprecise.